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# Indian Standard SPECIFICATION FOR SODIUM ALUMINATE

(First Revision)

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BUREAU OF INDIAN STANDARDS MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG NEW DELHI 110002

# Indian Standard SPECIFICATION FOR SODIUM ALUMINATE

# (First Revision)

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# Indian Standard SPECIFICATION FOR SODIUM ALUMINATE

(First Revision)

## O. FOREWORD

- 0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 18 June 1984, after the draft finalized by the Inorganic Chemicals (Misc) Sectional Committee had been approved by the Chemical Division Council.
- **0.2** The major application of sodium aluminate is in connection with water treatment and is based on its property of forming flocculant precipitates of aluminium hydroxide which are capable of coagulating, entraining and absorbing finely divided suspended solids and coloured materials. In addition, it possesses the property of coagulating colloidal magnesium hydroxide. Thus, sodium aluminate forms a useful adjunct to the limesoda softening process, particularly when magnesium is also present in water. It is also used in other industries where aluminium is required in an alkaline form or an alkaline coagulant is needed, for example, for printing on fabrics, manufacture of lake colours, sizing of paper, and hardening of building stones, and as filler in soaps.
- **0.3** Sodium aluminate is also used in conjunction with sulphate of alumina in the clarification of water supply in water works and in industries using river water. This process is known as double coagulation process.
- **0.4** This standard was originally prepared in 1967 with a view to helping the producers to manufacture a product of acceptable quality, and in laying down the requirements of the material, due consideration had been given to the indigenous production.
- **0.5** This standard has been revised incorporating new limits for iron oxide, silica and moisture as the Committee felt that these elements may affect those products where sodium aluminate is used as a raw material. The methods of estimation for iron oxide and silica have also been modified. This revision incorporates Amendment No. 1 issued earlier.
- **0.6** This standard contains clause **3.1** which calls for agreement between the purchaser and the supplier.

0.7 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS: 2-1960\*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

#### 1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for sodium aluminate.

# 2. REQUIREMENTS

- 2.1 Description The material shall be a white granular mass. It absorbs moisture and carbon dioxide rapidly when exposed to atmosphere and results in caking.
- 2.2 Stability Ten grams of the material when dissolved in 100 ml of freshly distilled water and allowed to stand for 24 hours in a closed bottle, shall not show appreciably larger flocculation or precipitation than that present in the beginning.
- 2.3 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of the table.
- 2.4 Moisture Content The moisture content shall be 4.0 percent by mass, maximum when tested according to the method given in Appendix B.

#### 3. PACKING AND MARKING

- 3.1 Packing The material shall be packed in mild steel black plate drums with polyethylene liner or as agreed to between the purchaser and the supplier. As the material has a tendency to absorb moisture and carbon dioxide from the atmosphere which results in caking the exposure of the material direct to air shall be avoided.
- 3.2 Marking The packages shall be marked with the following information:
  - a) Name of the material;
  - b) Name of the manufacturer or his recognized trade-mark, if any;

<sup>\*</sup>Rules for rounding off numerical values ( revised ).

- c) Mass of the material in the package; and
- d) Identification in code or otherwise to enable the batch of manufacturer to be traced from the records.

TABLE 1 REQUIREMENTS FOR SODIUM ALUMINATE ( Clause 2.3 )

| SL<br>No.    | CHARACTERISTIC  | REQUIREMENT  | METHOD OF TEST<br>( REF TO CL NO.<br>IN APPENDIX A ) |
|--------------|---|--------------|--|
| (1)          | <b>(2</b> )   | (3)          | <b>(4</b> )  |
| i)           | Caustic alkalinity (as Na2O), percent by mass, Min  | <b>4</b> 0·0 | A-3  |
| ii)          | Water-soluble aluminate (as Al <sub>3</sub> O <sub>8</sub> ), percent by mass, <i>Min</i> | <b>52</b> ·0 | A- <b>4</b>  |
| iii)         | Iron oxide (as Fe <sub>2</sub> O <sub>2</sub> ), parts per million, Max                   | 100          | <b>A-</b> 5  |
| i <b>v</b> ) | Matter insoluble in water, percent by mass, Max   | 1.2          | Λ-6  |
| v)           | Silica (as SiO <sub>2</sub> ), parts per million, Max                                     | 200          | A-7  |
| vi)          | Sulphates (as Na <sub>2</sub> SO <sub>4</sub> ), percent by mass, Max                     | 0.2          | A-8  |
| vii)         | Chlorides (as NaCl), percent<br>by mass, Max  | 0.2          | A-9  |
| viii)        | Carbonates (as Na <sub>2</sub> CO <sub>3</sub> ), percent by mass, Max                    | 1.6          | A-10   |
| ix)          | Lead (as Pb), parts per million, Max  | 50           | Λ-11   |
| x)           | Arsenic (as $As_2O_3$ ), parts per million, $Max$   | 8            | A-12   |

# 3.2.1 The packages may also be marked with the ISI Certification Mark.

Note — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

#### 4. SAMPLING

4.1 The procedure for drawing representative samples of the material, the number of tests to be made on the samples and the criteria for conformity of the lot to the requirements of this specification, shall be as given in Appendix C.

# APPENDIX A

(Clause 2.3, and Table 1)

#### METHODS OF TEST FOR SODIUM ALUMINATE

#### A-1. QUALITY OF REAGENTS

**A-1.1** Unless specified otherwise, pure chemicals and distilled water ( see IS: 1070-1977\*) shall be used in tests.

Note — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

#### A-2. PREPARED SOLUTION

A-2.1 Dissolve about 5 g of the accurately weighed material in carbon dioxide-free water. Make up the volume to 500 ml in a volumetric flask and filter the solution. This solution shall be the prepared solution to be used in tests.

# A-3. DETERMINATION OF CAUSTIC ALKALINITY (AS Na<sub>2</sub>O)

# A-3.1 Reagents

- A-3.1.1 Standard Hydrochloric Acid 0.5 N.
- **A-3.1.2** Phenolphthalein Indicator Dissolve 0.1 g in 100 ml of 60 percent rectified spirit.
  - **A-3.1.3** Barium Chloride Solution approximately 10 percent (m/v).
- A-3.2 Procedure Take 100 ml of the prepared solution in a conical flask and add 20 ml of barium chloride (neutralized) solution. Titrate it against standard hydrochloric acid using phenolphthalein as indicator.

<sup>\*</sup>Specification for water for general laboratory use ( second revision ).

#### A-3.3 Calculation

Caustic alkalinity (as Na<sub>2</sub>O), percent by mass 
$$= \frac{3 \cdot 10 \times V \times N}{M}$$

where

V == volume in ml of standard hydrochloric acid required for titration,

N = normality of standard hydrochloric acid, and

M =mass in g of the material present in the aliquot.

# A-4. DETERMINATION OF WATER-SOLUBLE ALUMINATE (AS $Al_2O_3$ )

A-4.0 Principle — Water-soluble aluminates are determined by precipitating the material with ammonium hydroxide twice, igniting and weighing as  $R_2O_3$ . The mass of oxides due to iron and titanium is deducted from it to calculate the mass of  $Al_2O_3$ .

#### A-4.1 Reagents

- A-4.1.1 Ammonium Chloride solids.
- A-4.1.2 Concentrated Nitric Acid See IS: 264-1976\*.
- A-4.1.3 Dilute Ammonium Hydroxide approximately 9 N.
- **A-4.1.4** Dilute Hydrochloric Acid -1:4(v/v).
- A-4.1.5 Ammonium Nitrate Solution approximately 2 percent (made neutral with ammonium hydroxide to methyl red or phenol red).
  - A-4.1.6 Methyl Red Indicator Solution 0.15 g in 500 ml of water.
- A-4.2 Procedure Take 25 ml of the prepared solution and dilute to 100 ml. Add 5 g of ammonium chloride and 3 to 4 ml of nitric acid. Boil the solution for a few minutes. Add 2 to 3 drops of methyl red solution. Add ammonium hydroxide dropwise, with stirring, until the colour of the solution changes to distinct yellow. Boil for one minute, filter through a filter paper (Whatman No. 41 or its equivalent) and wash the precipitate three times with hot ammonium nitrate solution. Transfer the precipitate to the original beaker washing the filter paper with the dilute hydrochloric acid.

<sup>\*</sup>Specification for nitric acid ( second revision ).

- A-4.2.1 Add hot dilute hydrochloric acid sufficient to dissolve the precipitate and boil. Add methyl red indicator and make it faintly ammoniacal as before. Boil again for one minute and filter the precipitate as before. Wash the precipitate with hot ammonium nitrate solution until free from chlorides and transfer it with the filter paper to a crucible, previously ignited to constant mass. Dry and ignite at a low temperature until all carbon is burnt off; then complete the ignition at 1 100°C for one hour. Cool the residue in a desiccator over a good desiccant and weigh. Re-ignite it until constant mass is attained.
- A-4.2.2 Determine the percentage of aluminium oxide by deducting from it the percentage of iron oxide as obtained under A-5.

# A-5. DETERMINATION OF IRON OXIDE (AS Fe<sub>2</sub>O<sub>3</sub>)

**A-5.0 Principle** — Iron oxide is determined colorimetrically using o-phenanthroline.

## A-5.1 Reagents

- **A-5.1.1** Standard Iron Solution (10  $\mu$ g'ml) Dissolve 0.491 l g of ferrous ammonium sulphate [(FeSO<sub>4</sub>), (NH<sub>4</sub>)<sub>2</sub> SO<sub>4</sub>, 6H<sub>2</sub>O)] in water. Add 10 ml of l:1 sulphuric acid and dilute with water to 1 000 ml. Pipette out 50 ml of the solution and dilute with water to 500 ml. One millilitre of this solution is equivalent to 0.01 mg of iron (as Fe<sub>2</sub>O<sub>3</sub>).
  - **A-5.1.2** Sulphuric Acid 1:1.
  - **A-5.1.3** Hydroxylamine Hydrochloride Solution 10 percent (m/v).
- A-5.1.4 o-Phenanthroline Solution Dissolve 0.1 g of o-phenanthroline in 2 ml of methanol and dilute to 100 ml with water and store in a dark bottle with a ground-glass stopper.
  - A-5.1.5 Sodium Acetate Solution 2 M.
- **A-5.2 Procedure** Take 10, 20, 30, 40 and 50 ml aliquots of standard iron solution in separate 100 ml beakers. Adjust the pH to 2 to 3 using 1:1 sulphuric acid and then between 3 to 4 using sodium acetate solution. Add 2 ml of hydroxylamine hydrochloride solution and 5 ml of o-phenanthroline solution. Dilute with water the contents of each beaker to 100 ml. Determine the absorbance after 30 minutes against reagent blank at 510 nm using a spectrophotometer. Plot a calibration curve of absorbance against concentration of 1.0  $\mu$ g/ml to 5  $\mu$ g/ml as obtained by following the above dilution steps using the standard iron solution.

A-5.2.1 Dissolve in water 2 g of the material, accurately weighed, and dilute with water to 100 ml. Take 10 ml aliquot in a 100 ml beaker. Adjust the pH to 2 to 3 using 1:1 sulphuric acid and then between 3 to 4 using sodium acetate solution. Add 2 ml of hydroxylamine hydrochloride solution and 5 ml of o-phenanthroline solution. Dilute with water the contents of the beaker to 100 ml. Determine the absorbance after 30 minutes against reagent blank at 510 nm using a spectrophotometer. Read the concentration for the absorbance determined using the calibration curve.

#### A-5.3 Calculation

Iron oxide (as  $Fe_2O_3$ ), percent by mass =  $\mu g/ml$  of  $Fe_2O_3$  from the calibration curve  $\times$  0.05

#### A-6. DETERMINATION OF MATTER INSOLUBLE IN WATER

A-6.1 Procedure — Weigh accurately 10 g of the material and dissolve by stirring in 100 ml of water. Filter through a sintered glass crucible. Wash the residue with hot water till it is free from alkali. Dry the residue at 13°C + 2°C to constant mass.

#### A-6.2 Calculation

Matter insoluble in water, percent by mass 
$$\frac{100 \times m}{M}$$

where

m = mass in g of the dried residue, andM = mass in g of the material taken for the test.

# A-7. DETERMINATION OF SILICA (AS SiO<sub>2</sub>)

**A-7.0 Principle** — Silica is determined colorimetrically using ammonium molybdate.

## A-7.1 Reagents

**A-7.1.1** Standard Silica Solution (  $10 \mu g/ml$ ) — Dissolve 0.314 g of dried sodium fluosilicate (  $Na_2SiF_6$ ) in hot water and dilute to 500 ml. Pipette out 50 ml aliquot and dilute with water to 1 000 ml. One millilitre of this solution is equivalent to 0.01 mg of silica ( as  $SiO_2$  ).

**A-7.1.2** Sulphuric Acid — 1:1 and 10 M.

**A-7.1.3** Ammonium Molybdate Solution — 10 percent (m|v|).

- A-7.1.4 Reducing Agent 0.7 g of sodium sulphite and 0.15 g of 1-amino-2 naphthol-4 sulphonic acid in 10 ml of water, dissolved in 9 g of sodium metabisulphite in 70 ml of water.
  - **A-7.1.5** Boric Acid 5 percent (m/v).
  - A-7.1.6 Hydrofluoric Acid 45 percent.
- A-7.2 Procedure Take 10, 20, 30, 40 and 50 ml aliquots of standard silica solution in separate 500 ml plastic beakers. Adjust the  $\rho$ H to 2.0 with 1:1 sulphuric acid. Add 50 ml of boric acid followed by exact addition to 4 drops of hydrofluoric acid. Add again 50 ml of boric acid and keep in a constant temperature bath at 40°C for 20 minutes. Add 4 ml of ammonium molybdate solution and keep at 48°C for further 20 minutes. Remove the beakers and add 20 ml of 10 M sulphuric acid and 5 ml of reducing agent. Dilute with water the contents of each beaker to 200 ml. Determine the absorbance after 15 minutes against reagent blank at 650 nm using a spectrophotometer. Plot a calibration curve of absorbance against concentration of 0.5  $\mu$ g/ml to 2.5  $\mu$ g/ml as obtained by following the above dilution steps using the standard silica solution.
- A-7.2.1 Dissolve in water 0.5 g of the material, accurately weighed, and dilute with water to 500 ml. Take 10 ml aliquot and dilute with water to 100 ml. Further, take 20 ml of this aliquot in a beaker and adjust the pH to 2.0 with 1:1 sulphuric acid. Transfer the contents to a 500 ml plastic beaker. Add 50 ml of boric acid followed by exact addition of 4 drops of hydrofluoric acid. Add again 50 ml of boric acid and keep in a constant temperature bath at 40°C for 20 minutes. Add 4 ml of ammonium molybdate solution and keep at 40°C for further 20 minutes. Remove the beaker and add 20 ml of 10 M sulphuric acid and 5 ml of reducing agent. Dilute with water the contents of the beaker to 200 ml. Determine the absorbance after 15 min against reagent blank at 650 nm using a spectrophotometer. Read the concentration for the absorbance determined using the calibration curve.

#### A-7.3 Calculation

Silica (as  $SiO_2$ ), percent by mass =  $\mu g/ml$  of  $SiO_2$  from the calibration curve  $\times$  10

# A-8. DETERMINATION OF SULPHATES (AS Na2SO4)

A-8.0 Principle — Sulphates are determined by precipitation with barium chloride solution.

# A-8.1 Reagents

A-8.1.1 Barium Chloride Solution — approximately 10 percent.

A-8.1.2 Concentrated Hydrochloric Acid - See IS: 265-1976\*.

A-8.2 Procedure — Take 50 ml of the prepared solution in a beaker. Acidify with concentrated hydrochloric acid so that all the aluminium hydroxide precipitates dissolve and add, with stirring, 10 ml of hot barium chloride solution slowly. Boil gently for 5 minutes and keep it for 2 hours on a steam bath. Allow the contents of the beaker to stand for 4 hours and filter through a filter paper (Whatman No. 42 or its equivalent). Wash the precipitates with hot water till it is free from chlorides. Transfer the residue in a porcelain crucible and ignite it again at 800°C, cool and weigh till constant mass is obtained.

A-8.2.1 As the maximum limit for lead is 50 parts per million, no correction is necessary for lead content:

#### A-8.3 Calculation

Sulphates (as Na<sub>2</sub>SO<sub>4</sub>), 
$$=\frac{60.86 \times m}{M}$$

where

m =mass in g of the residue of barium sulphate obtained, and M =mass in g of the material taken for the test ( see A-7.2 ).

A-8.4 Alternatively, the sulphate content may also be estimated in accordance with 3 of IS: 9506-1980†.

# A-9. DETERMINATION OF CHLORIDES (AS NaCl)

A-9.0 Principle — Chlorides are determined by titration with silver nitrate solution using potassium chromate as indicator. When the entire chloride is precipitated as silver chloride, chromate ions combine with silver to form red silver chromate.

# A-9.1 Reagents

- A-9.1.1 Dilute Nitric Acid approximately 5 N.
- A-9.1.2 Sodium Bicarbonate solid.
- **A-9.1.3** Potassium Chromate Solution approximately 5 percent (m/v).
- A-9.1.4 Standard Silver Nitrate Solution 0.1 N.

A-9.2 Procedure — Take 25 ml of the prepared solution in a 250-ml conical flask. Just acidify with dilute nitric acid. Neutralize the excess of the acid by the addition of sodium bicarbonate. Add 1 ml of potassium

<sup>\*</sup>Specification for hydrochloric acid ( second revision ).

<sup>†</sup>Method for determination of sulphate (volumetric).

chromate solution and titrate with standard silver nitrate solution with vigorous shaking until the first permanent colour deviation from the pure yellow of the suspension is obtained. Carry out a blank titration, using the same quantities of other reagents in the same total volume of the reaction mixture.

#### A-9.3 Calculation

Chlorides (as NaCl), percent by mass = 
$$\frac{5.846 \times N(V_1 - V_2)}{M}$$

where

 $\mathcal{N}$  = normality of the standard silver nitrate solution,

 $V_1$  = volume in ml of standard silver nitrate solution required for titration,

 $V_2$  = volume in ml of standard silver nitrate solution required for blank, and

M =mass in g of the material contained in the aliquot.

# A-10. DETERMINATION OF CARBONATES (AS Na<sub>2</sub>CO<sub>3</sub>)

A-10.0 Principle — Carbon dioxide is generated by adding dilute hydrochloric acid in vacuum and is absorbed in an excess of alkali which is back titrated with a standard hydrochloric acid.

# A-10.1 Apparatus

A-10.1.1 Kjeldahl Flask — 300-ml capacity.

A-10,1,2 Drechsel Battle - provided with two taps.

A-10.1.3 Vacuum Pump — to give pressure less than 10 cm of mercury.

A-10.1.4 Tap Funnel

# A-10.2 Reagents

A-10.2.1 Methyl Orange Indicator — Dissolve 0.01 g of methyl orange in 100 ml of water.

**A-10.2.2** Sodium Hydroxide Solution — 1 N.

A-10.2.3 Barium Chloride Solution - 10 percent.

A-10.2.4 Phenolphthalein Indicctor — Dissolve 0'l g of phenolphthalein in 100 ml of 60 percent rectified spirit.

A-10.2.5 Dilute Hydrochloric Acid - 5 N, 1 N and 0.1 N.

A-10.3 Procedure — Take 5 g of the accurately weighed material in a 300-ml Kjeldahl flask A and add about 100 ml of water and two drops of methyl orange indicator. Assemble the apparatus as shown in Fig. 1. Provide the flask A with a rubber bung through which passes a short bend of wide capillary tubing and a tap funnel B reaching nearly to the bottom of the flask. Place 20 ml of sodium solution and 1 ml of phenolphthalein indicator in a Drechsel bottle C provided with taps E and F. Connect this bottle to the Kjeldahl flask and evacuate the whole assembly to less than 10 cm mercury pressure using a vacuum pump. Introduce 40 ml of 5 N hydrochloride acid through the tap funnel into the flask ( the precipitated aluminium hydroxide should redissolve and excess acid should be present as indicated by methyl orange), avoiding the admission of air. Keep about 1 ml of acid in the funnel to prevent air leaking during subsequent operation. Heat the flask over a small flame until the outlet capillary tube becomes hot, shaking the Drechsel bottle occasionally to absorb the carbon dioxide. The solution in Drechsel bottle should remain pink. Fill the flask A with boiling water through the tap to flush out any traces of carbon dioxide held back in the flask. Shake the Drechsel bottle and disconnect it after removing the vacuum by opening the outlet tap X. Titrate the excess sodium hydroxide with 1 N hydrochloric acid (phenolphthalein already present) so as not to overstep the end point. Boil off carbon dioxide. It is better to complete this titration near the end point with 0.1 N hydrochloric acid till the solution is just colourless. Add two drops of methyl orange indicator and excess of 1 N hydrochloric acid. Shake well and titrate with sodium hydroxide solution. Carry out the blank in the same way.

#### A-10.4 Calculation

Carbonates (as 
$$Na_2CO_3$$
),  $V_1 - (V_3 - V_2) \times 0.053 \times 100$  percent by mass

where

 $V_1 =$ excess of standard hydrochloric acid (1 N) added to the solution,

 $V_2$  volume of standard sodium hydroxide solution consumed in the titration,

V<sub>3</sub> - volume of standard hydrochloric acid (1 N) used for blank titration, and

M - mass in g of the material taken for the test.

# A-11. DETERMINATION OF LEAD (As Pb)

A-11.0 Principle — Lead is determined by comparing the colour produced by the material with hydrogen sulphide solution against that produced by a standard lead solution.

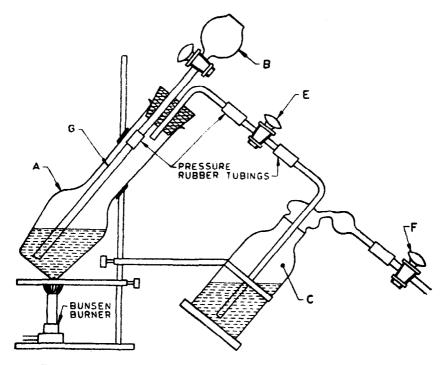


Fig. 1 Apparatus for the Determination of Carbonates

# A-11.1 Apparatus

A-11.1.1 Nessler Cylinders — 50 ml capacity.

# A-11.2 Reagents

- A-11.2.1 Concentrated Hydrochloric Acid See IS: 265-1976\*.
- A-11.2.2 Standard Lead Solution Dissolve 1.60 g of lead nitrate in water and make up the volume to 1 000 ml. Transfer exactly 10 ml of the solution to a volumetric flask and dilute it again with water to 1 000 ml mark. One millilitre of this solution contains 0.01 mg of lead (as Pb). The dilute solution shall be freshly prepared.

A-11.2.3 Acetic Acid - approximately 1 N.

<sup>\*</sup>Specification for hydrochloric acid ( second revision ).

# A-11.2.4 Hydrogen Sulphide Solution - saturated.

A-11.3 Procedure — Weigh 10 g of the material and add 15 ml of water. Add 6 ml of concentrated hydrochloric acid and evaporate to dryness on a water-bath. Dissolve the residue in water and transfer this solution to a Nessler cylinder. Dilute it with water to 30 ml and add 1 ml of acetic acid and 10 ml of hydrogen sulphide solution. In the second Nessler cylinder, carry out a control test using 5 ml of standard lead solution and the same quantities of other reagents. Dilute the contents of each cylinder to 50 ml and shake well. Compare the colour produced in the two cylinders.

A-11.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced with the material is not greater than that produced in the control test.

# A-12. DETERMINATION OF ARSENIC (AS As<sub>2</sub>O<sub>3</sub>)

A-12.1 Procedure — Dissolve 1.0 g of material in the minimum amount of water. Carry out the test for arsenic as prescribed in IS: 2088-1971\*, using for comparison a stain obtained with 0.008 mg of arsenic trioxide (as  $As_2O_3$ ).

A-12.2 The limit prescribed in Table 1 shall be taken as not having been exceeded if the length and intensity of the stain is not greater than that produced in the control sample.

# APPENDIX B

(Clause 2.4)

#### **B-1. DETERMINATION OF MOISTURE**

**B-1.1 Procedure** — Take 5 g of the accurately weighed material in a tared squat type bottle and dry it at  $130^{\circ}\text{C} \pm 2^{\circ}\text{C}$  in an oven till constant mass is obtained. This temperature has been specified in order to reduce the time of the heating.

#### B-1,2 Calculation

Moisture, percent by mass = 
$$\frac{(M_1 - M_2) \times 100}{M}$$

where

 $M_1 = \text{mass in g of material taken, and}$ 

 $M_2$  = mass in g of the material after heating in the oven.

<sup>\*</sup>Methods for determination of arsenic (first revision).

# APPENDIX C

( Clause 4.1 )

#### SAMPLING OF SODIUM ALUMINATE

### C-1. GENERAL REQUIREMENTS OF SAMPLING

C-1.0 Reference for general requirements of sampling, reference may be made to IS: 8883 (Part 1)-1978\*.

#### C-2. SCALE OF SAMPLING

- **C-2.1 Lot** In any consignment, all the containers of the same size and drawn from the same batch of manufacture shall be grouped together to constitute a lot.
- C-2.2 For ascertaining the conformity of the material in the lot to requirements of this specification, tests shall be carried out for each lot separately. The number of containers to be selected for this purpose shall depend upon the size of the lot and shall be in accordance with Table 2.
- **C-2.3** The containers shall be selected at random from the lot. To ensure randomness of selection, a random number table may be used. In case such a table is not available, the following procedure may be adopted:

Starting from any container in the lot, count them as  $1, 2, 3, \ldots$  up to r and so on in one order. Every rth container thus counted shall be withdrawn to constitute the sample, where r is the integral part of N|n, N being the number of containers in the lot and n the number of containers to be selected.

#### C-3. PREPARATION OF SAMPLES

C-3.1 From each of the selected containers draw with an appropriate sampling instrument a representative portion of the material not less than 300 g. Divide it into three equal parts of about 100 g and transfer each to a clean and moisture-proof sample container which shall be closed air-tight ensuring that during this whole procedure the material and the sample are exposed to the atmosphere as little as possible. Each of these three samples shall be as individual sample representing that particular container.

<sup>\*</sup>Methods of sampling chemicals and chemical products: Part 1 General requirements and precautions.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING (Clause C-2.2)

| NUMBER OF CONTAINERS<br>IN THE LOT | NUMBER OF CONTAINERS<br>TO BE SELECTED |
|------------------------------------|--|
| ${\mathcal N}$                     | n                                      |
| (1)                                | (2)                                    |
| Up to 50                           | 3                                      |
| 51 to 100                          | 4                                      |
| 101 to 300                         | 5                                      |
| 301 and above                      | 7                                      |

- C-3.2 There will be three sets of individual samples. Each set will contain a sample representing each selected container. One of these sets shall be marked for the purchaser, another for the supplier and the third shall be used as a referee set.
- C-3.3 The referee set consisting of individual samples representing the selected containers shall bear the seal of both the purchaser and the supplier or their representatives. They shall be kept at a place and under conditions agreed to between the parties. The referee set shall be used in the case of dispute only.

#### C-4. NUMBER OF TESTS

- **C-4.1** Tests for the determination of caustic alkalinity, water-soluble aluminate and moisture content shall be conducted on each of the individual samples in a set.
- **C-4.2** The samples on which the individual tests are carried out, the average  $\bar{X}$  and range (R) shall be calculated:

where

$$\bar{X} = \frac{\text{Sum of test results}}{\text{Number of tests}}$$

R = Difference between maximum and minimum testresults

The lot shall be declared as conforming to the requirements of the individual sample if  $\bar{X} = 0.6R$  is greater than or equal to the minimum value specified and  $\bar{X} + 0.6R$  is less than or equal to the maximum value specified in Table 1.

#### C-5. CRITERIA FOR CONFORMITY

- C-5.1 For Individual Samples The lot shall be declared as conforming to the requirements of caustic alkalinity, water-soluble aluminate and moisture content if each of the individual samples separately satisfies the relevant requirements.
- C-5.2 For Composite Sample For declaring the conformity of the lot to the requirements of the characteristics tested on the composite sample, the test result for each characteristic shall satisfy the corresponding requirements of this specification.

#### ( Continued from page 2)

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